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IS 6267 (1971): Silver Cyanide and Silver Potassium Cyanide for Electroplating [CHD 5: Electroplating Chemicals and Photographic Materials]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 6267 - 1971
(Reaffirmed 2008)

Indian Standard

**SPECIFICATION FOR
SILVER CYANIDE AND SILVER POTASSIUM
CYANIDE FOR ELECTROPLATING**

(Second Reprint DECEMBER 1997)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR SILVER CYANIDE AND SILVER POTASSIUM CYANIDE FOR ELECTROPLATING

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AMENDMENT NO. 1 FEBRUARY 1985

TO

IS: 6267-1971 SPECIFICATION FOR

SILVER CYANIDE AND SILVER POTASSIUM

CYANIDE FOR ELECTROPLATING

(Page 4, clause 2.2) — Add the following after 2.2:

'2.2.1 For use in special electroplating applications like electrical and electronic engineering industries, the requirements of silver cyanide and silver potassium cyanide, permissible limits of copper (as Cu) and chloride (as Cl) and any other impurities, may be as agreed to between the purchaser and the manufacturer.

2.2.1.1 For guidance, copper (as Cu) may not exceed 0.005 percent by mass and chloride (as Cl) 0.02 percent by mass.

2.2.1.2 The methods of determination of these requirements shall be as agreed to between the purchaser and the manufacturer.'

[Page 4, Table 1, *Sl No.* (iii)] — Add the following new requirement under respective columns:

(1)	(2)	(3)	(4)	(5)
iv) Copper (as Cu), percent by mass, <i>Max</i>	0 05	0.05	A-5'	

(Page 7, clause A-4.1) — Add the following after A-4.1:

'A-5. DETERMINATION OF COPPER IN SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

A-5.1 Procedure — Weigh 2.0 g accurately of the material. Add 10 ml nitric acid and 10 ml sulphuric acid. Titrate the mixture to sulphuric acid fumes for decomposition of cyanides in a hood. Cool the above acidic solution and add 20 ml water. Wash the dropper and dish. Collect all washings. Precipitate all silver by adding about 5 to 10 ml saturated solution of sodium chloride. Filter silver chloride through Whatman's filter paper No. 40 and wash with water. Collect all the filtrates and make just alkaline with 25 percent caustic soda and then make acidic with acetic acid. Add 1 g of potassium iodide and shake for 2 to 3 minutes. Keep the flask aside for 5 minutes in the dark.

Titrate the liberated iodine with $\frac{N}{100}$ sodium thiosulphate solution using starch as indicator when the blue colour disappears.

A-5.2 Calculation — Copper as (Cu), percent by mass = $6.35 \times \frac{VN}{M}$

where

V = volume in ml of standard sodium thiosulphate solution,

N = normality of standard thiosulphate solution, and

M = mass in g of the sample taken for the test.'

(CDC 43)

Indian Standard

SPECIFICATION FOR SILVER CYANIDE AND SILVER POTASSIUM CYANIDE FOR ELECTROPLATING

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 23 September 1971, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 Silver is normally electrodeposited from a solution containing essentially a double cyanide of silver and an alkali metal, additional alkali cyanide (free cyanide) and other alkaline compounds. The solution is usually produced from silver cyanide and silver potassium cyanide. The anodes used are usually stainless steel or silver. Requirements of silver anodes are covered in IS : 1959-1961*.

0.3 In the formulation of this standard, due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in electroplating chemicals industry in this country. This has been met by deriving assistance from BS 1361 : 1966 'Silver anodes and silver salts for electroplating' issued by the British Standards Institution.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for silver cyanide and silver potassium cyanide for electroplating.

*Specification for silver anodes for electroplating.

†Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description

2.1.1 Silver Cyanide — The material shall be in the form of creamy white crystals, free from dirt, foreign matter and visible impurities and shall correspond essentially to formula AgCN .

2.1.2 Silver Potassium Cyanide — The material shall be in the form of white powder free from dirt, foreign matter and visible impurities and shall correspond essentially to the formula $\text{KAg}(\text{CN})_2$.

2.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR SILVER CYANIDE AND SILVER POTASSIUM CYANIDE FOR ELECTROPLATING

SL No.	CHARACTERISTIC	REQUIREMENTS		METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Silver Cyanide	Silver Potassium Cyanide	
(1)	(2)	(3)	(4)	(5)
i)	Silver (asAg), percent by mass (dry basis), <i>Min</i>	80.0	54.0	A-2
ii)	Matter insoluble in aqueous solution of potassium cyanide, percent by mass, <i>Max</i>	0.1	—	A-3
iii)	Matter insoluble in distilled water, percent by mass, <i>Max</i>	—	0.1	A-4

3. SAFETY PRECAUTIONS IN HANDLING SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

3.1 Since silver cyanide and silver potassium cyanide are highly poisonous, extreme caution is necessary in handling the same. Useful information on this subject is given in Appendix B.

4. PACKING AND MARKING

4.1 Packing — Unless otherwise agreed to between the purchaser and the vendor the salts shall be supplied in airtight containers which shall not have any reaction with the contents.

AMENDMENT NO. 1 FEBRUARY 1985

TO

IS : 6267-1971 SPECIFICATION FOR

SILVER CYANIDE AND SILVER POTASSIUM

CYANIDE FOR ELECTROPLATING

(*Page 4, clause 2.2*) — Add the following after **2.2**:

2.2.1 For use in special electroplating applications like electrical and electronic engineering industries, the requirements of silver cyanide and silver potassium cyanide, permissible limits of copper (as Cu) and chloride (as Cl) and any other impurities, may be as agreed to between the purchaser and the manufacturer.

2.2.1.1 For guidance, copper (as Cu) may not exceed 0.005 percent by mass and chloride (as Cl) 0.02 percent by mass.

2.2.1.2 The methods of determination of these requirements shall be as agreed to between the purchaser and the manufacturer.'

[*Page 4, Table 1, Sl No. (iii)*] — Add the following new requirement under respective columns:

(1)	(2)	(3)	(4)	(5)
iv) Copper (as Cu), percent by mass, <i>Max</i>		0.05	0.05	A-5'

(*Page 7, clause A-4.1*) — Add the following after **A-4.1**:

'A-5. DETERMINATION OF COPPER IN SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

A-5.1 Procedure — Weigh 2.0 g accurately of the material. Add 10 ml nitric acid and 10 ml sulphuric acid. Titrate the mixture to sulphuric acid fumes for decomposition of cyanides in a hood. Cool the above acidic solution and add 20 ml water. Wash the dropper and dish. Collect all washings. Precipitate all silver by adding about 5 to 10 ml saturated solution of sodium chloride. Filter silver chloride through Whatman's filter paper No. 40 and wash with water. Collect all the filtrates and make just alkaline with 25 percent caustic soda and then make acidic with acetic acid. Add 1 g of potassium iodide and shake for 2 to 3 minutes. Keep the flask aside for 5 minutes in the dark.

Titrate the liberated iodine with $\frac{N}{100}$ sodium thiosulphate solution using starch as indicator when the blue colour disappears.

A-5.2 Calculation — Copper as (Cu), percent by mass = $6.35 \times \frac{VN}{M}$

where

V = volume in ml of standard sodium thiosulphate solution,

N = normality of standard thiosulphate solution, and

M = mass in g of the sample taken for the test.'

(CDC 43)

4.2 Marking — The containers shall be marked with the following:

- a) Name of material and its net weight;
- b) Name of manufacturer and recognized trade-mark, if any;
- c) Lot number and date of manufacture; and
- d) The word 'POISON' and the appropriate symbol (*see* IS : 1260-1958*).

4.2.1 The containers may also be marked with the Standard Mark.

4.2.2 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The method of preparing representative samples of the material and the criteria for conformity to this standard shall be as prescribed in Appendix G.

APPENDIX A

(*Clause 2.2, and Table 1*)

ANALYSIS OF SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†), shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF SILVER IN SILVER CYANIDE AND SILVER POTASSIUM CYANIDE

A-2.1 Reagents

A-2.1.1 *Concentrated Nitric Acid* — (*see* IS : 264-1968‡).

*Code of symbols for labelling dangerous goods.

†Specification for water, distilled quality (*revised*).

‡Specification for nitric acid (*first revision*).

A-2.1.2 Ferric Ammonium Sulphate Solution — 20 percent (w/v).

A-2.1.3 Potassium Thiocyanate Solution — 0.1 N, prepared by dissolving 9.717 0 g of potassium thiocyanate in 1 000 ml of water.

A-2.1.4 Hydrogen Cyanide Detection Papers — Prepare as required by dipping strips of filter paper in a mixture of equal volumes of solutions A and B prepared as follows:

Solution A — 0.1 percent (w/v) o-tolidine acetate — Dissolve 0.64 g of o-tolidine in 5 ml of 50 percent (w/v) glacial acetic acid solution and dilute to 1 000 ml with water.

Solution B — 0.3 percent (w/v) copper acetate solution in water.

A-2.2 Procedure — Weigh accurately about 1 g of the sample and transfer to a 250-ml conical flask. Add 20 ml of concentrated nitric acid and boil in a fume cupboard until a clear solution is obtained, and test for complete destruction of cyanide with hydrogen cyanide detection papers. Dilute to approximately 100 ml with water, add 2 ml of ferric ammonium sulphate solution and titrate with standardized potassium thiocyanate solution till the colour changes to red-brown tint.

A-2.3 Calculation

$$\text{Silver (as Ag), percent by mass} = \frac{V \times N \times 10.79}{W}$$

where

V = volume in ml of standard potassium thiocyanate solution consumed,

N = normality of potassium thiocyanate solution, and

W = mass in g of the sample taken for the test.

A-3. DETERMINATION OF MATTER INSOLUBLE IN AQUEOUS SOLUTION OF POTASSIUM CYANIDE

A-3.1 Reagent

A-3.1.1 Potassium Cyanide Solution — 10 percent (w/v).

A-3.2 Procedure — Weigh accurately about 10 g of silver cyanide and dissolve in 100 ml of warm potassium cyanide solution. Filter the solution through a tared sintered glass crucible (G No. 3). Wash thoroughly with hot water and dry the crucible at 110°C. Weigh again and express the mass of the residue as percentage of the mass of the material taken for the test.

A-4. DETERMINATION OF MATTER INSOLUBLE IN DISTILLED WATER

A-4.1 Procedure — Weigh accurately about 10 g of silver potassium cyanide and dissolve it in 100 ml of water. Filter the solution through a tared sintered glass crucible (G No. 3). Wash thoroughly with hot water and dry at 110°C. Weigh again and express the mass of the residue as percentage of the mass of the material taken for the test.

APPENDIX B

(*Clause 3.1*)

**SAFETY PRECAUTIONS FOR HANDLING SILVER CYANIDE
AND SILVER POTASSIUM CYANIDE****B-1. SAFETY PRECAUTIONS**

B-1.1 As silver cyanide and silver potassium cyanide are highly poisonous, they should never be touched with unprotected hands; gloves should always be worn during sampling operations, and during crushing, goggles fitted with a face cloth should also be worn.

B-1.2 Solutions containing cyanide should never be pipetted by mouth suction. An evacuating bulb should always be used to draw the liquid into the pipette. A burette may also be used for drawing measured quantity of the solution.

B-1.3 On account of the highly poisonous character of hydrogen cyanide all operations involving the decomposition of cyanides should be conducted in a well-ventilated fume cupboard.

B-2. HYDROCYANIC ACID AND CYANIDE POISONING

B-2.1 Symptoms — The symptoms are giddiness, staggering and insensibility accompanied by panting respiration, and followed by profound collapse with convulsions. The action is extremely rapid.

B-2.2 First Aid

B-2.2.1 Remove patient from the cause of trouble, for example, fumes, etc, and take him to fresh air. Make the patient lie down, keep him warm and do not allow him to move more than necessary.

B-2.2.2 If breathing has ceased, apply artificial respiration.

NOTE — Mouth to mouth respiration should not be attempted.

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B-2.2.3 Administer amyl nitrite. This is purchased in the form of small ampoules and one of these is broken and held under the nose so that the patient will inhale the vapour. It should be administered for 15 to 30 seconds every 2 to 3 minutes.

NOTE — Amyl nitrite is sensitive to light and warmth and should, therefore, be kept in the dark at a temperature less than 15°C. All ampoules should be discarded every 2 years.

B-2.2.4 If available, administer oxygen through a face mask and call for a qualified medical practitioner.

B-2.3 Antidote

B-2.3.1 The following antidote has been found useful when cyanide is swallowed:

- a) *Solution A* — Dissolve 158 g of ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and 3 g of citric acid in 1 000 ml of water. The solution should be regularly inspected and replaced wherever any deterioration occurs.
- b) *Solution B* — Dissolve 60 g of anhydrous sodium carbonate in 1 000 ml of water.

B-2.3.1.1 Take 50 ml from each of solutions A and B and keep in separate 175-ml wide-necked bottles with a polyethylene closure. Mark the bottles as 'Cyanide Antidote A' and 'Cyanide Antidote B'. Both the bottles should bear the legend 'Mix the whole contents of bottles A and B and administer the mixture'.

B-2.4 Medical Treatment

B-2.4.1 The treatment consists of the injection into the blood-stream of sodium nitrite and sodium thiosulphate and should be carried out only by a qualified medical practitioner. Details are as follows:

- a) Intravenous injection of 0.3 g of sodium nitrite dissolved in 10 ml of sterile distilled water. This should be given slowly at the rate of 2.5 ml to 5 ml per minute.
- b) Immediately following this and through the same needle an intravenous injection of 25 g of sodium thiosulphate dissolved in 50 ml of sterile distilled water is given at the same rate. Leakage of material outside the vein should be avoided.

B-2.4.2 A temporary improvement is not a criterion of recovery. If the symptoms persist or recur after an hour a second injection of the two substances should be given. It is suggested that electroplating shops using cyanide should keep a supply of these two substances in ampoule form and two sterilized syringes, one with a total capacity of 10 ml and

the other with a total capacity of 50 ml, together with particulars of the treatment (as above).

B-2.4.3 Since it is unlikely that the average general practitioner or hospital would have such material ready for use, should the patient be sent to hospital these materials should accompany him in the ambulance, and during the journey the first-aid procedures already described should be continued.

B-2.4.4 Some patients may respond to the first-aid treatment alone, but in many cases it will be advisable, if not necessary, to give the intravenous injection. This should be done as soon as possible, and in any case it is desirable that it should be administered within 15 minutes. It is, therefore, essential that if medical help cannot immediately be obtained, the patient should be conveyed without delay to the nearest hospital.

APPENDIX C

(Clause 5.1)

SAMPLING OF SILVER CYANIDE AND SILVER POTASSIUM CYANIDE FOR ELECTROPLATING

C-1. GENERAL REQUIREMENTS OF SAMPLING

C-1.0 In drawing, preparing, storing and handling samples, the safety precautions prescribed in Appendix B shall be strictly followed.

C-1.1 The sampling implements and the sample containers shall be clean and dry.

C-1.2 Each sample container shall be sealed airtight after filling and marked with full details of sampling.

C-2. SCALE OF SAMPLING

C-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot.

C-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

C-2.3 In order to ensure randomness of selection random number tables shall be used. For random selection procedures, guidance may be had from IS:4905-1968*.

*Methods of random sampling.

TABLE 2 SCALE OF SAMPLING
(Clause C-2.2)

LOT SIZE (THE NUMBER OF CONTAINERS)	SAMPLE SIZE
Up to 25	3
26 „ 50	4
51 „ 150	5
151 „ 300	6
301 and above	8

C-3. INDIVIDUAL AND COMPOSITE SAMPLE

C-3.1 From each of the containers selected according to C-2.2 a representative portion of the material sufficient for the tests shall be withdrawn. These samples representing each of the selected containers are termed as individual samples.

C-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken. Such portions shall be thoroughly mixed to give a composite sample.

C-3.3 The material constituting each of the individual sample as well as the composite sample shall be stored separately with full identification particulars.

4. NUMBER OF TESTS

C-4.1 Test for silver content shall be conducted on individual samples.

C-4.2 Test for the remaining characteristics shall be done on the composite sample.

C-5. CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples — For silver content the test results shall be noted and their mean (\bar{X}) and the range (R), being the difference between the maximum and minimum of test results, shall be computed. For declaring the conformity of the lot in respect of silver content, ($\bar{X} - 0.6R$) shall be greater than or equal to the minimum specified in Table 1.

C-5.2 For declaring the conformity of the lot to the requirements of all other characteristics, the test results of the composite sample shall satisfy the relevant requirements.

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